

FIG. 1

PROVIDE AN ALUMINA POWDER PRECURSOR

ADD ABOUT 1-10% MAGNESIA POWDER
PRECURSOR AND 1-10% TITANIA POWDER
PRECURSOR TO THE ALUMINA POWDER
PRECURSOR TO MAKE A GREEN POWDER
PRECURSOR

MIX THE GREEN POWDER PRECURSOR

PRESSING A GREEN BODY FROM THE GREEN
POWDER PRECURSOR

REMOVING RESIDUAL MOISTURE AND
ORGANIC MATERIAL FROM THE GREEN BODY

FIRING THE GREEN BODY IN AIR TO ABOUT
CONE 13

2090E0" DBD2600T

FIGURE 2

Some experimental data for a typical low-fired high alumina body

| | |
|--|--|
| Alumina Content | Low Firing Alumina Composition |
| Acid Resistant | Silica-Free Formulation |
| Starting Grain Size | $\frac{1}{2} \mu\text{m}$ |
| Grain Growth | 1-3 μm (avg. 1.2 +/- 0.3 μm) |
| Material Preparation | Ball Milling, Spray Drying, Dry Pressing |
| Firing | Performed in Normal Atmosphere, Fiber-Lined Furnace, Fired to Cone 13 |
| Specific Gravity | 3.8 + (@ 1350°C) |
| Water Absorption | 0 |
| Thermal Conductivity @ 25°C (cal/cm ² /sec/°C) | 0.05 |
| Thermal Expansion @ 300 - 1000°C (°C) | 8.3×10^{-6} |
| Thermal Expansion Rate | Substantially Uniform – There is Negligible Thermal Expansion Mismatch Between the Matrix and Second Phase |
| Toughness, K _{IC} | 4-5 Mpa·m ^½ |
| Rockwell Hardness (45N) | >80 |
| Hardness (GPa) | 16 |
| Elastic Modulus | 365 Gpa |
| Flexural Strength (kpsi) | 47 |
| Compressive Strength (kpsi) | >300 |
| Tensile Strength (kpsi) | 25 |
| Dielectric Strength (v/mil) | 250 (open atmosphere) |
| Dielectric Constant @ 1 kHz | 8.2 |
| Dissipation Factor @ 1 kHz | 0.001 |
| Loss Factor @ 1 kHz | .01 |
| T _e (°C) | >900 |
| Volume Resistivity @ 25°C | >10 ¹⁴ |
| Surface Finish as Fired: | 10 rms ± 2 (typical sample) |
| Ground: | 5 rms ± 1 (typical sample) |

FIGURE 3

Long-Term Corrosion Study of Typical Low-Temperature High-Alumina (LTHA):

| <u>MATERIAL</u> | <u>WEIGHT LOSS</u> |
|-----------------|--------------------|
| ZTA | -70 percent |
| SiC/SiC | -30 percent |
| LTHA Sample | -30 percent |

**Materials Corrosion Test:
Independent Test**

Weight loss in mg/dm²/day

46.7% Hydrofluoric (HF) acid @ 25°C

| | <u>5 day immersion</u> | <u>35 day immersion</u> (30 after 5) |
|---|------------------------|---|
| SiC-Silica Free | 1.00 | 1.00 |
| ZrO ₂ -Toughened | 1110.00 | 1070.00 ⁽¹⁾ |
| Al ₂ O ₃ - 99.9% | 1.92 | 2.26 |
| LTHA Sample (Membrane-approx. 36% porosity) | 1.0 | 0.16 ⁽²⁾ |
| LTHA Sample (Solid) | 1.64 | 0.09 |

NOTE: Weight loss is listed in mg/dm²/day rounded to nearest 0.01g.

- (1): Approximately 2/3 of the coupon was destroyed in 35 days of testing.
(2): This is a rather severe test in that the surface area is approx. 36% greater than the normal as tested.

Materials Corrosion Test:

50% H₃PO₄ @ 25°C

| | <u>Cum. Mg/dm² (approx.)</u> | |
|-------------|---|-----------|
| | 24 Hours | 120 Hours |
| AD90 | 5.35 | 9.65 |
| AD94 | 2.72 | 5.00 |
| AD96 | 4.82 | 12.54 |
| ADO96 | 5.61 | 11.59 |
| AD99.5 | 6.75 | 10.26 |
| TTZ | 0.88 | 3.33 |
| LTHA Sample | 1.66 | 2.02 |



30% NaOH @ 25°C

Cum. Mg/dm² (approx.)

| | 24 Hours | 120 Hours |
|-------------|----------|-----------|
| AD90 | 24.98 | 51.15 |
| AD94 | 15.24 | 32.27 |
| AD96 | 2.13 | 6.10 |
| ADO96 | 11.59 | 14.61 |
| AD99.5 | 8.23 | 12.20 |
| TTZ | 0.61 | 0.61 |
| LTHA Sample | 1.72 | 2.01 |

NOTE: Weight loss is mg/dm²/day, rounded to nearest 0.01g.

Materials Corrosion Test:

Weight loss in mg/cm²/day

| | <u>60% H₃PO₄ @ 60°C</u> | <u>30% NaOH @ 60°C</u> |
|---|---|------------------------|
| A479 Al ₂ O ₃ (90%) | 0.15 | 0.28 |
| A479SS Al ₂ O ₃ (99.5%) | 0.07 | 0.12 |
| 3NaI ₂ O ₃ (99.9%) | 0.02 | 0.00 |
| LTHA Sample | 0.00 | 0.00 |

NOTE: Weight loss is mg/cm²/day, rounded to nearest 0.01g.

High Alumina Corrosion Test:

Independent Test

| <u>CORROSIVE SOLUTION</u> | <u>HCl</u> | <u>HNO₃</u> | <u>H₂SO₄</u> |
|--------------------------------------|----------------------|------------------------|------------------------------------|
| <u>MATERIAL</u> | <u>% WEIGHT LOSS</u> | | |
| Product of Manufacturer A: | | | |
| 85% Al ₂ O ₃ | 0.066 | 0.076 | 0.066 |
| 96% Al ₂ O ₃ | 0.081 | 0.087 | 0.200 |
| LTHA Sample | (No Detectable Loss) | | |
| Product of Manufacturer B: | | | |
| 99.5% Al ₂ O ₃ | 0.217 | 0.163 | 0.216 |

PROCEDURES

1. Check the initial weight (approximately 5 grams)
2. Immerse into high concentration acid/base solutions
3. Dilute with 50 volume % of distilled water
4. Boil for an hour, and let soak overnight
5. Check the final weight
6. Calculate percent weight loss

$$\% \text{ LOSS} = (\text{INITIAL WEIGHT} - \text{FINAL WEIGHT}) / \text{INITIAL WEIGHT}$$

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FIGURE 4

Summary of Typical Low-Temperature High-Alumina Material Thermal Spray Features:

| | | <u>APPLICATION METHOD</u> | |
|---|---------------------------|---------------------------|-----------------------|
| | | <u>Subsonic Plasma</u> | <u>HVOF</u> |
| Deposit Efficiency (DE) | | 85+% | 75+% |
| Lay-Down Rate (surface speed = 1200 ipm) | | 4+mil/pass | 1+mil/pass |
| Hardness (as applied) | Rockwell Vickers | 90+ 1,100+ | 94+ 1300+ |
| Surface Roughness (as applied) | | 200 – 300 μ in. | 60 – 80 μ in. |
| Dielectric Strength | | 450+v/mil | 950+v/mil |
| Bond Strength | | 10,000 – 12,000 psi | |
| Porosity (by optical microscopy) | Interconnected Surface | | 0% < 1/2 μ in. |

Properties of LTHA Coating

| | | <u>APPLICATION METHOD</u> | |
|--|---------------------------|---------------------------|---------------------------|
| | | <u>Subsonic Plasma*</u> | <u>HVOF**</u> |
| Deposit Efficiency (DE) | | 85.5% (82 - 91) | 74% (65 - 83) |
| Lay-Down Rate (mil/pass) (surface speed = 1200 ipm) | | 3.5 (2 - 4.2) | 0.98 (0.87 – 1.03) |
| Hardness (as applied) | Rockwell Std. Dev. | 90.1 (88.9 – 91.7) 1.4 | 94.3 (94.1 – 94.7) 0.4 |
| Surface Roughness (as applied) | | 200 – 300 μ in. | 60 – 80 μ in. |
| Dielectric Strength | | 450+v/mil | 950+v/mil |
| Bond Strength | | 10,000 – 12,000 psi | |
| Porosity (by optical microscopy) | Interconnected Surface | | 0% < 1/2 μ in. |

* These averages are based on 132 sample coupons, randomly picked for testing out of approximately 30 spray runs containing 8 – 10 coupons per run.

** These averages are based on 30 sample coupons, randomly picked for testing out of approximately 10 spray runs containing 8 – 10 coupons per run.

() Indicates ranges of high and low values.

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FIGURE 5**Tensile Test Data for 6061 Matrix Composites at 15v% (Test One)**

(NOTE: This test conducted with non-spherical grain)

| Material | H.T. Condition | Elastic Modulus (Msi) | Yield Strength (ksi) | Ultimate Strength (ksi) | Strain at Failure (%) |
|--|-------------------|-----------------------------|----------------------------|-------------------------------|-----------------------------|
| 6061 (avg.) | T - 6 | 9.9 | 40 | 45 | 10 |
| LTHA Sample | T - 6 | 12.9 | 53 | 65 | 7 |
| Tabular Al ₂ O ₃ | T - 6 | 12.9 | 53 | 65 | 7 |

Tensile Test Data for 7093 Matrix Composites at 15v% (Test Two)

| Material | H.T. Condition | Elastic Modulus (Msi) | Yield Strength (ksi) | Ultimate Strength (ksi) | Strain at Failure (%) |
|--|-------------------|-----------------------------|----------------------------|-------------------------------|-----------------------------|
| 7093 (avg.) | T - 6 | 10.3 | 92 | 95.6 | 13.4 |
| LTHA Sample | T - 6 | 13.1 | 80 | 85 | 0.9 |
| Tabular Al ₂ O ₃ | T - 6 | 13.4 | 58 | 67 | 4.2 |
| B ₄ C | T - 6 | 14.6 | 84 | 98 | 2.6 |

Tensile Test Data for 7093 Matrix Composites at 10v% (Test Three)

| Material | H.T. Condition | Elastic Modulus (Msi) | Yield Strength (ksi) | Ultimate Strength (ksi) | Strain at Failure (%) |
|-----------------------------|-------------------|-----------------------------|----------------------------|-------------------------------|-----------------------------|
| 7093 (avg.) | T - 6 | 10.3 | 92 | 95.6 | 13.4 |
| LTHA Sample | T - 6 | 12.2 | 82.5 | 90.0 | 3.7 |
| B ₄ C (96 samp.) | T - 6 | 13.2 | 89 | 96.7 | 4.1 |

Thermal Expansion Coefficient: 10 ppm/°F (Al = 13 ppm/°F)

Friction and Water Data:

| Composite ID | Coefficient of Friction | | Volume loss from Block (10 ⁻³ cu cm) |
|---------------------|-------------------------|--------|--|
| | Start | Finish | |
| 20v% SiC/2124 | .096 | .119 | 6.34 |
| 25v% SiC/2124 | .101 | .123 | 6.23 |
| 30v% SiC/2124 | .098 | .119 | 4.15 |
| 20v% SiC/7091 | .101 | --- | 6.31 |
| SPF-251 Std Coating | .141 | .129 | 13.11 |

Test Results of 7093/Al₂O₃/xyp Composite

| Sample | Vol. % | YS 0.2% (ksi) | UTS (%) | Elongation (Msi) | Modulus |
|--|--------|------------------|------------|---------------------|---------|
| Baseline T - 6 | 0 | 85.7 | 96.5 | 22.4 | 10.6 |
| Tabular Al ₂ O ₃ T - 6 | 15 | 58.0 | 67.0 | 4.2 | 14.1 |
| Medialox C25CR | 10 | 25.1 | 33.9 | 2.43 | --- |
| Duralox DF500 | 10 | 36.5 | 48.7 | 7.56 | --- |
| Baikalo GE6 | 10 | 24.9 | 37.0 | 4.53 | --- |
| LTHA Sample Lot 1 T - 6 | 15 | 84.3 | 86.2 | 0.9 | 13.5 |
| LTHA Sample Lot 2 T - 6 | 10 | 82.5 | 89.6 | 3.7 | 12.2 |

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